

PROJECT NUMBER: 2520
PROJECT TITLE : Flavor Research
PROJECT LEADER : Y. Houminer
PERIOD COVERED : December, 1990

I. FLAVOR RELEASE TECHNOLOGY

- A. Objective: To investigate the synthesis and pyrolysis of various flavor release systems for use in new or improved products.
- B. Results: We continue to explore the synthesis and pyrolysis of various menthol release agents. The reaction of di-O-acetyl-L-tartaric anhydride with menthol was carried out at 145°C. After workup, a foamy hygroscopic solid was obtained which was recrystallized from hexane to give a solid with a sharp melting point. A sample has been sent for elemental analysis and NMR.

Several other menthol release agents were obtained. Dimethyl oxalate was synthesized and purified by flash chromatography. A pure sample of the material was obtained and has been submitted for NMR and elemental analysis. Purification of the mono-menthyl diglycolate has been completed. Samples have been submitted for NMR and elemental analysis.

A large sample of beta-menthyl itaconate was purified to give 54gm of pure ester. An additional 10 grams of calcium beta-menthyl itaconate has been prepared for use by Flavor Development in spray application for subjective evaluation.

Dr. L. Harwood from Oxford University has informed us that his group has discovered that they can get about 70% distilled yields of MIC using sodium hydride instead of potassium hydride. They are planning to use the same conditions to prepare isopropenyl chloroformate.

We have reported in the past that phenethyl β -D-glucopyranose reacts with menthyl chloroformate in dioxane to give 66% yield of the 6-O-carbonate. We have recently discovered that methyl α -D-glucopyranoside reacts with menthyl chloroformate in pyridine to give a mixture of compounds: the 6-O- and 2-O-carbonates, and a third component which is believed to be the 2,6-O-dicarbonate. The difference in selectivity can be attributed to the solvent effect as we have demonstrated before in the reaction of glucose with menthyl chloroformate.

We continue to explore the grafting of the trans-2,3-cyclic carbonate of Aromatek 245 to other carbohydrates using xylital as a model compound. Several experiments were carried out. It was observed that in the presence of triethylamine, the cyclic carbonate group was extremely sensitive to moisture, and the compound decomposed spontaneously to Aromatek-245. In the presence of pyridine, however, the cyclic carbonate group was extremely stable and only very little decomposition was observed. On the other hand, the reactivity of the cyclic carbonate with xylitol was very slow, even in the presence of triethylamine. So far most of the experiments resulted in decomposition of the cyclic carbonate, or recovery of starting material.

We continue to investigate other reaction conditions.

Samples of both CR-2950 and CR-2951 have been delivered to Flavor Development for evaluation. Both compounds are designed to release α -hexylcinnamaldehyde.

II. FILTER MATERIALS AND PAPER TECHNOLOGY SUPPORT

- A. **Objective:** To find outside suppliers for large quantities of new filter materials and inorganic paper additives.
- B. **Results:** R.S.A. has completed the laboratory scale development work on the production of magnesite by hydrothermal carbonylation. Both Reheis magnesium hydroxide powder and 30% paste have been converted to magnesite in about 24 hrs. The paste gave 99% and the powder 98% magnesite respectively. Samples will be arriving the week of December 17th and evaluation of the material will be carried out by J. Fournier and B. Rogers.

Based on the above results, R.S.A. feels that they will be able to conduct this chemistry in their large pressure reactor. They will be supplying approximate cost, size, and available production dates later this month. Final production will not be run until the lab samples have been evaluated and the projected cost reviewed.

Work continues at Forest Products Labs (FPL) on the large scale acetylation of cellulose. In a recent run (batch #6) the degree of acetylation was 0.44, which is about half of the target degree of acetylation.

Analytical research has completed the analysis of the papers made from the FPL samples. Results show that the papers are uniformly lower (between 15 to 30%) in acetyl content than the original samples. NMR analysis confirms these results. Bob Rogers is evaluating the paper making process to see if the loss may be there.

Dr. David Giachardi (Director of R&D, Courtaulds) and Dr. David Randall (Director of R&D, Courtaulds Acetate) met with PM scientists in late November. Courtaulds is willing to enter into a cooperative business agreements with PM. The two primary areas will be Fibril systems and partially acetylated cellulose systems. Agreements are being prepared.

III. FLAVOR CHEMISTRY

- A. **Objective:** To obtain flavors for subjective evaluation and odor profiling. To isolate and identify tobacco components which are sensorially significant.
- B. **Results:** We continue to prepare different flavors which are not commercially available for evaluation in the odor profile program. Isopropenyl pyrazine was prepared from ethylpyrazine to compare its odor character (popcorn-nutty) to the material previously prepared from acetylpyrazine. The odor character was found to be unrelated to the method of preparation, thus indicating the absence of any impurities in the product.

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2-Isopropenylpyridine was prepared from 2-ethylpyridine. Surprisingly its odor character is an intense green-floral, not popcorn nutty.

Free-radical alkylation of pyridine using n-valeric acid gave a number of butylated products. A similar reaction with thiazole gave no significant amount of product and only 15% recovered unreacted thiazole.

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